


**ICF International / Laboratory Data Consultants**

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**MEMORANDUM**

TO: Richard Hiatt, Remedial Project Manager  
 Site Cleanup Section 3, SFD-7-3

THROUGH: Rose Fong, ESAT Task Order Manager (TOM)  
 Quality Assurance (QA) Program, MTS-3

FROM: Doug Lindelof, Data Review Task Manager  
 Region 9 Environmental Services Assistance Team (ESAT)

ESAT Contract No.: EP-W-06-041  
 Technical Direction Form No.: 00405005

DATE: July 16, 2008

SUBJECT: Review of Analytical Data, Tier 3

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:	Carson River
Site Account No.:	09 R6 QB00
CERCLIS ID No.:	None Provided
Case No.:	None
SDG No.:	ACE-0801
Laboratory:	CEBAM Analytical, Inc.
Analysis:	Methyl Mercury
Samples:	55 Sediment and Soil Samples (see Case Summary)
Collection Date:	February 5 and 6, 2008
Reviewer:	Stan Kott, ESAT/Laboratory Data Consultants

This report has been reviewed by the EPA TOM for the ESAT contract, whose signature appears above.

If there are any questions, please contact Rose Fong (QA Program/EPA) at (415) 972-3812.

Attachment

SAMPLING ISSUES: ☐ Yes ☒ No



## Data Validation Report – Tier 3

Case No.: None  
SDG No.: ACE-0801  
Site: Carson River  
Laboratory: CEBAM Analytical, Inc.  
Reviewer: Stan Kott, ESAT/LDC  
Date: July 16, 2008

### I. CASE SUMMARY

#### Sample Information

Samples: CR-S01-A-SS-020608, CR-S01-B-SS-020608,  
CR-S01-C-SS-020608, CR-S01-D-SS-020608,  
CR-S01-E-SS-020608, CR-S01-F-SS-020608,  
CR-S02-A-SS-020608, CR-S02-B-SS-020608,  
CR-S02-C-SS-020608, CR-S02-D-SS-020608,  
CR-S02-E-SS-020608, CR-S03-A-SS-020608,  
CR-S03-B-SS-020608, CR-S03-C-SS-020608,  
CR-S03-D-SS-020608, CR-S03-E-SS-020608,  
CR-S03-F-SS-020608, CR-S04-A-SS-020608,  
CR-S04-B-SS-020608, CR-S04-C-SS-020608,  
CR-S04-D-SS-020608, CR-S04-E-SS-020608,  
CR-S05-A-SS-020608, CR-S05-B-SS-020608,  
CR-S05-C-SS-020608, CR-S05-D-SS-020608,  
CR-S05-E-SS-020608, CR-S05-F-SS-020608,  
CR-S06-A-SS-020608, CR-S06-B-SS-020608,  
CR-S06-C-SS-020608, CR-S06-D-SS-020608,  
CR-S06-E-SS-020608, CR-S07-A-SS-020508,  
CR-S07-B-SS-020508, CR-S07-C-SS-020508,  
CR-S07-D-SS-020508, CR-S07-E-SS-020508,  
CR-S07-F-SS-020508, CR-S08-A-SS-020508,  
CR-S08-B-SS-020508, CR-S08-C-SS-020508,  
CR-S08-D-SS-020508, CR-S08-E-SS-020508,  
CR-S09-A-SS-020508, CR-S09-B-SS-020508,  
CR-S09-C-SS-020508, CR-S09-D-SS-020508,  
CR-S09-E-SS-020508, CR-S09-F-SS-020508,  
CR-S10-A-SS-020608, CR-S10-B-SS-020608,  
CR-S10-C-SS-020608, CR-S10-D-SS-020608,  
and CR-S10-E-SS-020608

Concentration and Matrix: Low Concentration Sediment and Soil  
Analysis: Methyl Mercury  
Method: Method 1630 / CEBAM Method CA-0024-B  
Collection Date: February 5 and 6, 2008  
Sample Receipt Date: February 8, 2008  
Preparation Date: February 9 through 12, 2008  
Analysis Date: February 12, 13, and 14, 2008

#### Field QC

Equipment Blanks (EB): Not Provided  
Background Samples (BG): Not Provided  
Field Duplicates (D1): CR-S01-B-SS-020608 and CR-S01-F-SS-020608  
Field Duplicates (D2): CR-S03-B-SS-020608 and CR-S03-F-SS-020608  
Field Duplicates (D3): CR-S05-A-SS-020608 and CR-S05-F-SS-020608

Field Duplicates (D4): CR-S07-B-SS-020508 and CR-S07-F-SS-020508  
Field Duplicates (D5): CR-S09-C-SS-020508 and CR-S09-F-SS-020508

#### Laboratory QC

Method Blanks & Associated Samples: Method Blanks (MB) and samples listed above  
Matrix Spike (MS)/MS Duplicate (SD): CR-S01-B-SS-020608MS/SD,  
CR-S02-E-SS-020608MS/SD,  
CR-S05-A-SS-020608MS/SD,  
CR-S09-B-SS-020508MS/SD,  
and CR-S10-D-SS-020608MS/SD  
Duplicate: CR-S05-E-SS-020608 RE

Analysis: Methyl Mercury

<u>Analyte</u>	<u>Sample Preparation and Distillation Date</u>	<u>Analysis Date</u>
Methyl Mercury	February 9 through 12, 2008	February 13 and 14, 2008
Percent Solids	February 11, 2008	February 12, 2008

#### Sampling Issues

None.

#### Additional Comments

The following quality control (QC) information is not provided in the data package and could not be evaluated: method detection limit (MDL) study (see EPA Method 1630, Section 9.2.1), initial precision and recovery (IPR) data (see EPA Method 1630, Section 9.2.2), ongoing precision and recovery (OPR) data (see EPA Method 1630, Section 9.5), and quality control sample (QCS) data (see EPA Method 1630, Section 9.6).

The 0.614 ng/g methyl mercury result reported for sample CR-S02-E-SS-020608SD is not correct. The correct concentration as determined from the raw data is 0.631 ng/g on a dry weight basis. The corrected value is in bold face type and highlighted in the data spreadsheet.

The dry weight methyl mercury results for sample duplicate CR-S05-E-020608RE and all matrix spikes were calculated by ESAT using the percent solids data from the original samples. These changes are highlighted in bold face print in the data spreadsheet. No adverse effect on data quality is expected.

Laboratory Method CA-0021-B, Section 2.4.6 states that "Under the conditions described here, recoveries are not 100% efficient in recovering methyl mercury using both distillation or solvent extraction. Therefore results should be recovery corrected." However, recovery correction was not required in the approved field sampling and analysis plan based on the laboratory's expectation of MS/MSD recoveries close to 100%. It should be noted that although the overall mean MS recovery was 95.5%, the analytical batch MS recoveries for this project ranged from 73% to 111%, and relative percent differences (RPDs) for the MS duplicates ranged from 2% to 10%. The effect on data quality is not known.

Analytical results are listed in the data spreadsheet with qualifications. Definitions of data qualifiers used in the data spreadsheet are provided in Table 1B.

This report was prepared in accordance with the following documents:

- Region 9 Standard Operating Procedure 906, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Inorganic Data Packages*;
- *Method 1630 Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and CVAFS* (EPA-821-R-01-020, January 2001);
- *Method CA-0021-B, Determination of Methyl Mercury (MeHg) in Water by Distillation, (or Solvent Extraction), Aqueous Phase Ethylation, Tenax Trap collection, GC Separation and Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)* (CEBAM Analytical, Inc.);
- *Method CA-0024-B, Determination of Methylmercury (MeHg) in Sediments and Soil by GC/CVAFS after Acid Leaching / Solvent Extraction/Ethylation (Modified EPA 1630)* (CEBAM Analytical, Inc.); and
- *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, October 2004.

## II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

	<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
1.	Data Completeness	Yes	
2.	Sample Preservation and Holding Times	Yes	
3.	Calibration	No	C,D
	a. Initial		
	b. Initial and Continuing Calibration Verification		
4.	Blanks	Yes	
5.	Laboratory Control Sample (LCS)	Yes	
6.	Duplicate Sample Analysis	Yes	
7.	Matrix Spike Sample Analysis	Yes	
8.	Field Duplicate Sample Analysis	No	E
9.	Compound Identification	No	A
10.	Sample Quantitation	Yes	B
11.	Overall Assessment	Yes	

N/A = Not Applicable

## III. VALIDITY AND COMMENTS

- A. The following results are rejected and flagged "R" in the data spreadsheet because the identity of the methyl mercury could not be confirmed.

- Methyl mercury in samples CR-S01-A-SS-020608 and CR-S05-F-SS-020608

Method 1630 requires that the gas chromatograph run at least one minute beyond the point at which the diethyl mercury peak returns to baseline (see EPA Method 1630, Section 11.3.8). This was not done for the samples listed above; they were analyzed before the diethyl mercury peak for the previous run returned to baseline.

In addition, the chromatograms for the samples listed above have four peaks instead of three as described by the method. Since more than one sample was analyzed during the 20 minute data collection period, the methyl mercury peak could not be identified with certainty. (See Attachment A, sample numbers 1 and 28.)

- B. Results above the MDL but below the reporting limit (RL) (denoted with an "L" qualifier) are estimated and flagged "J" in the data spreadsheet.

*Results above the MDL but below the RL are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of quantitation.*

- C. The following results are estimated and flagged "J" in the data spreadsheet because the sample concentration exceeds the highest calibration standard concentration.

- Methyl mercury in samples CR-S07-C-SS-020508 (initial analysis), CR-S07-D-SS-020508, CR-S07-E-SS-020508, and CR-S10-A-SS-020608

The methyl mercury results for the samples listed above are from undiluted samples with concentrations that exceed the highest methyl mercury standard concentration analyzed. The samples listed above were not reanalyzed with a smaller aliquot. The results reported for methyl mercury in the samples listed above are considered quantitatively uncertain.

*The laboratory SOP specifies that if a sample concentration exceeds the highest calibration standard, a standard concentration greater than the highest sample concentration is analyzed or the sample is diluted and re-analyzed.*

- D. The following results are estimated and flagged "J" in the data spreadsheet because a closing continuing calibration verification (CCV) standard was not analyzed.

- Methyl mercury in samples CR-S06-D-SS-020608, CR-S06-E-SS-020608, CR-S07-A-SS-020508, CR-S07-B-SS-020508, CR-S07-C-SS-020508 (initial analysis), CR-S07-D-SS-020508, CR-S07-E-SS-020508, CR-S07-F-SS-020508, CR-S08-A-SS-020508, CR-S08-B-SS-020508, CR-S08-C-SS-020508, CR-S08-D-SS-020508, CR-S08-E-SS-020508, and CR-S09-A-SS-020508

A closing CCV was not analyzed for the samples listed above (see EPA Method 1630, Section 10.2). Results greater than or equal to the MDL are considered quantitatively uncertain.

*The laboratory SOP specifies that the laboratory must analyze a CCV standard at the beginning of the analytical run and at the end of the analytical day.*

- E. Relative percent differences (RPDs) of 40% and 67% were obtained for methyl mercury in the analysis of field duplicate pair (D3) samples CR-S05-A-SS-020608 and CR-S05-F-SS-020608 and field duplicate pair (D4) samples CR-S07-B-SS-020508 and CR-S07-F-SS-020508, respectively. Since sampling variability is included in the measurement, field duplicate results are expected to vary more than laboratory duplicates which have a  $\pm 35$  RPD criterion for precision. The effect on data quality is not known.

*The analysis of field duplicate samples is a measure of both field and analytical precision. The imprecision in the results of the analysis of the field duplicate pair may be due to the sample matrix, sample non-homogeneity, or poor sampling or laboratory technique.*





**TABLE 1B**

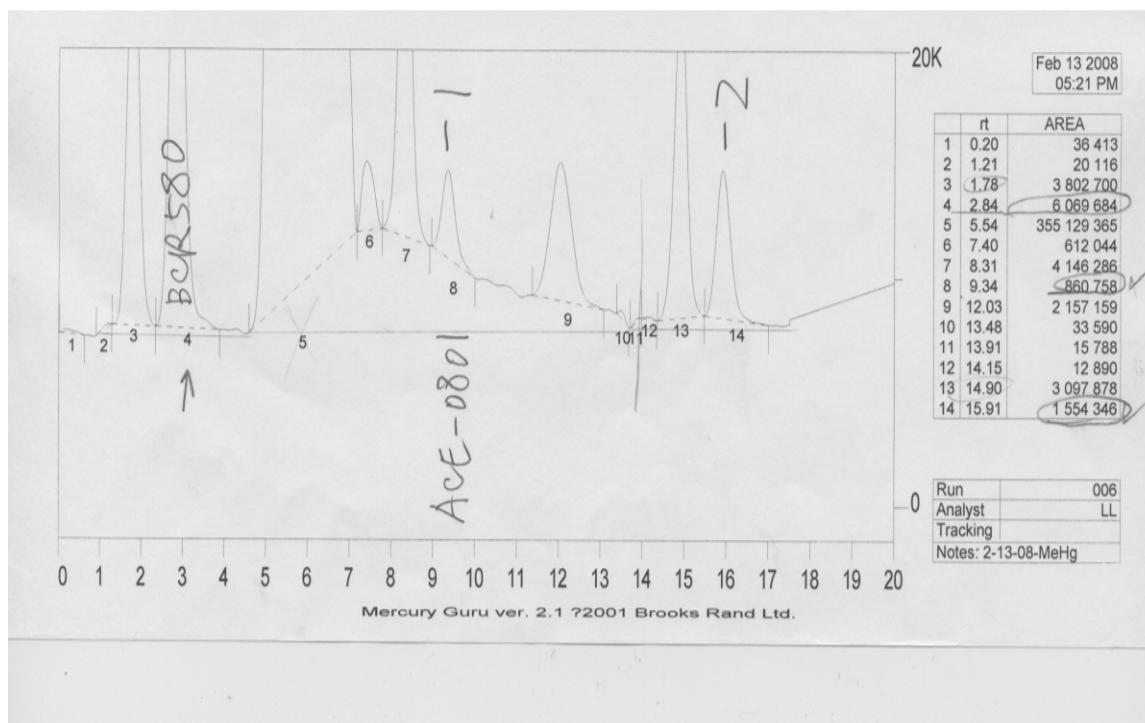
**DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW**

The definitions of the following qualifiers are prepared in accordance with the document *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, October 2004.

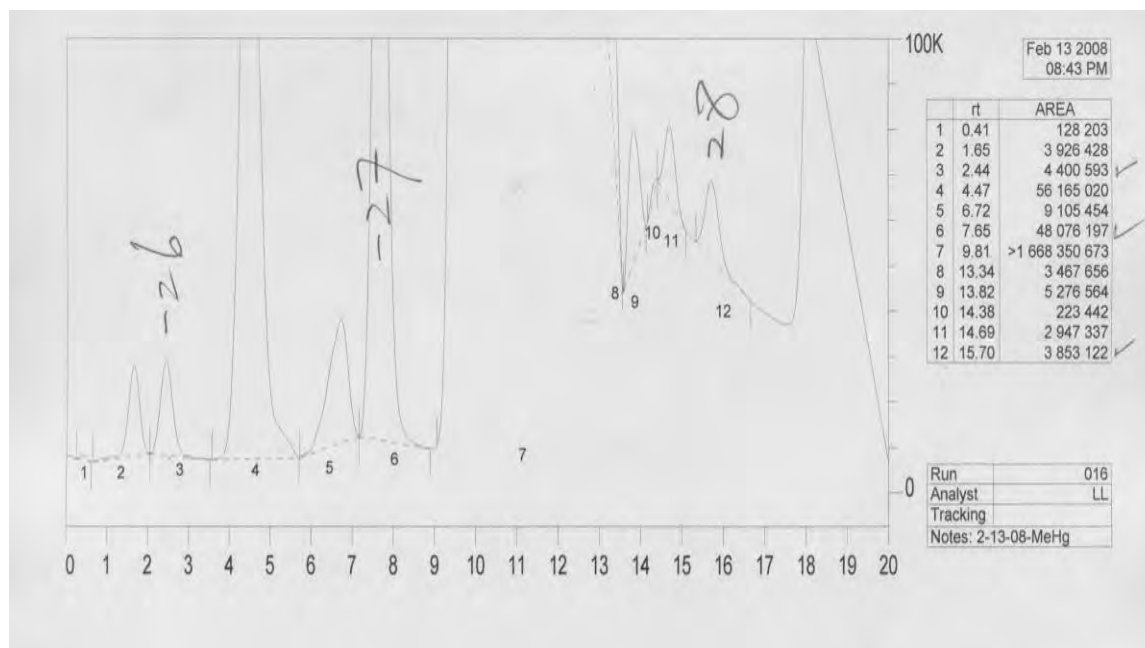
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.



# Attachment A



Sample CR-S01-A-SS-020608 (number 1)



Sample CR-S05-F-SS-020608 (number 28)



